Indian J. Phys. **79**(2), 143-146 (2005)

INTERNATIONAL JOURNAL
IJP

Setting up of a PIXE facility at FOTIA at BARC

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Received 31 March 2004, accepted 20 December 2004

Abstract : PIXE-(Proton Induced X-ray Emission) – an ideal technique for material characterization has been set up at the Folded Tandem Ion Accelerator (FOTIA) at BARC, Trombay. A separate beam line at 45° port in which a dedicated PIXE chamber has been installed is being used for PIXE studies. PIXE studies on gold standards were carried out using protons of energy 3.3 MeV. Caritage values of gold standards were obtained which agree well with the certified values. Single element standard solutions (100 μg/ml) for low-*Z* elements such as Cr, Mn, Fe, Co, Cu and Zn were analyzed by PIXE. Well resolved α and β components of the X-rays were seen with detection limits in the range of 0.8–4 μg/g.

Keywords : PIXE, FOTIA, gold standards, low-*Z* elements, detection limits.

PACS No. : 32.30.Rj

1. Introduction

Proton Induced X-ray Emission (PIXE) is a more sensitive technique than X-ray fluorescence due to higher excitation cross sections for excitation of lower atomic number elements [1] and easier availability of large flux of proton beam. Due to its higher cross sections for low energy elements, PIXE is useful in a number of applications for detecting low levels of trace elements in bio-sciences [2–4] as well as in material science [5,6], which have been carried out in our laboratory at Van de Graaff. A PIXE facility is now available and in constant use at the newly commissioned FOTIA (Folded Tandem Ion Accelerator) [7] at BARC. Gold standards of 22, 20, 18 and 14 carats was checked for Au/Ag ratios with protons of 3.3 MeV. In an attempt to check detector resolution and observe the *K* X-rays of low-*Z* elements ($20 > Z > 30$), thin targets of standard solutions of 1 mg/ml (100 ppm) were excited with 3 MeV protons delivered at the FOTIA accelerator at BARC, Trombay, and Mumbai.

2. Experimental details

Gold standards of 1 cm diameter and 0.5 mm thickness were mounted on an aluminium ladder and placed in the PIXE chamber (Figure 1) under a vacuum of the order of 10^{-6}. Solutions of low, *Z* elements such as Cr ($Z = 24$), Mn ($Z = 25$), Fe ($Z = 26$), Co ($Z = 28$), Cu ($Z = 29$), Zn ($Z = 30$) (100 μl) were micro pipetted on 3 microns thick Mylar backing and dried by slow heating under a infrared lamp. They were mounted on an aluminum ladder and enclosed in a PIXE chamber. In both cases, a collimated beam (5 mm dia) of protons of current 7 nA and energy 3.3 MeV was directed at the targets which

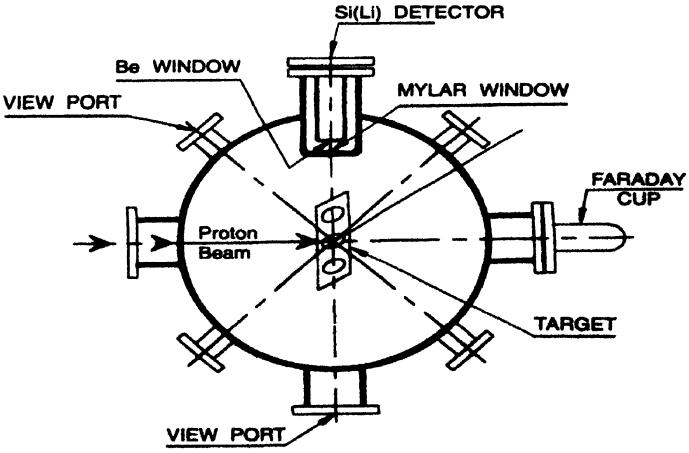

Figure 1. PIXE chamber.

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was placed at an angle of 45° to the beam direction and at an angle of 135° to the detector (Figure 2). The characteristic X-rays of the elements passes through a

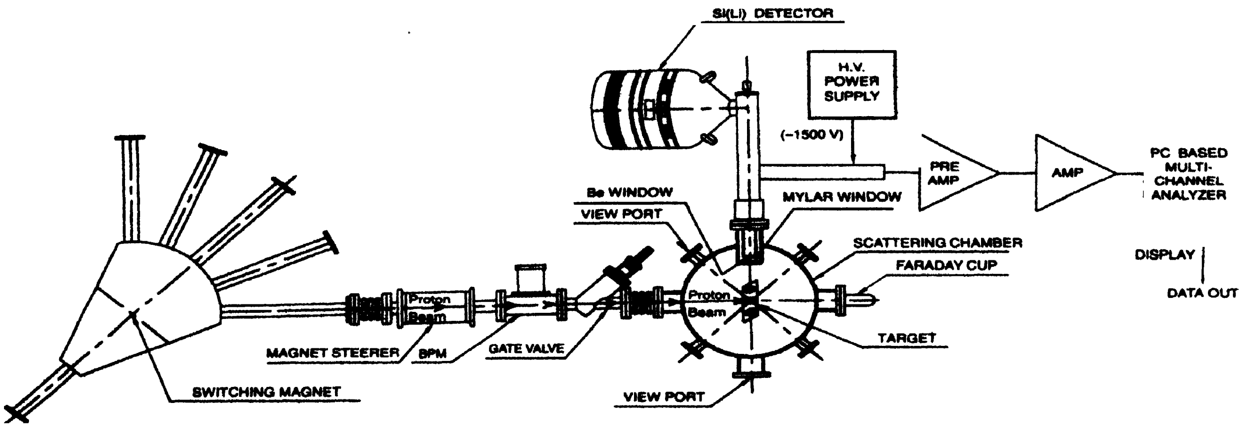

Figure 2. Experimental set-up for Proton Induced X-Ray Emission (PIXE) studies.

0.3 micron thickness Mylar window, a 10 cm air gap and then a 1 mil thin Be window to detected by the detector. The detector is kept at an angle of 90° to the beam direction. All samples were counted for a counting time of 1000 seconds. The X-ray spectra were stored and analyzed by PC-based MCA. The observed intensities were corrected for energy losses in Mylar window and self-absorption corrections.

3. Results and discussion

The PIXE X-ray spectra of Cr, Co, Mn, Cu and Zn elements are shown in Figures 3(a-e). The spectra show

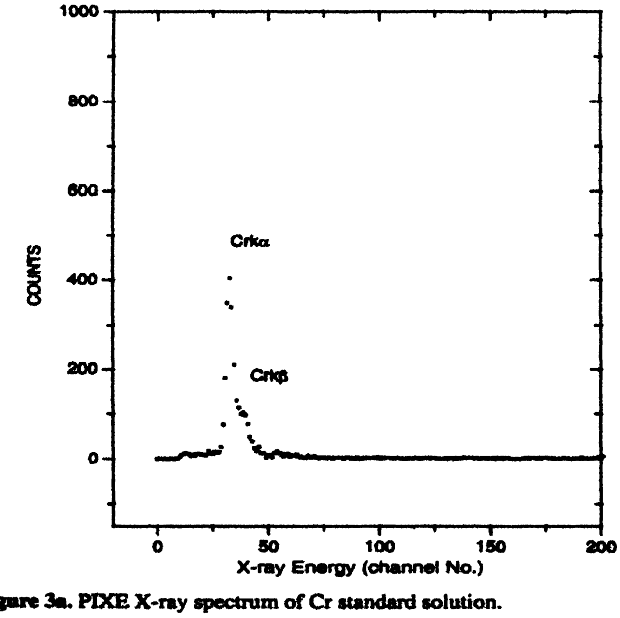

Figure 3a. PIXE X-ray spectrum of Cr standard solution.

well resolved lines of α and β components of K X-rays of all elements except for Cr and Mn. The α component of these elements were obtained from their respective

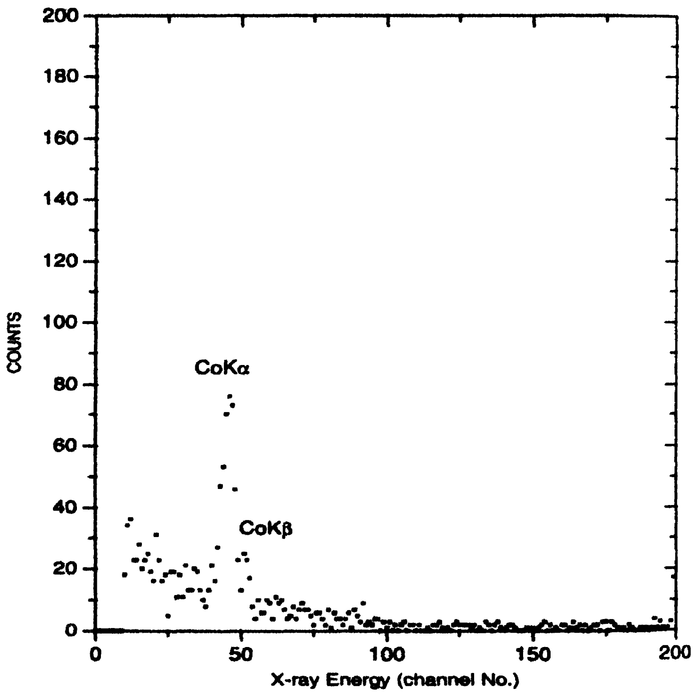

Figure 3b. PIXE X-ray spectrum of Co standard solution.

K_β/K_α ratios and intensities recorded. Table 1 gives the percentage values of Au and Ag in the gold standards and their comparison with certified values. Table 2 gives detection limits of the low-Z elements. The PIXE X-ray spectra show minimum background level.

Figure 4 gives an X-ray spectrum of 22-carat gold standard. The X-ray counts from the PIXE spectra were corrected for absorption effects and proton energy losses

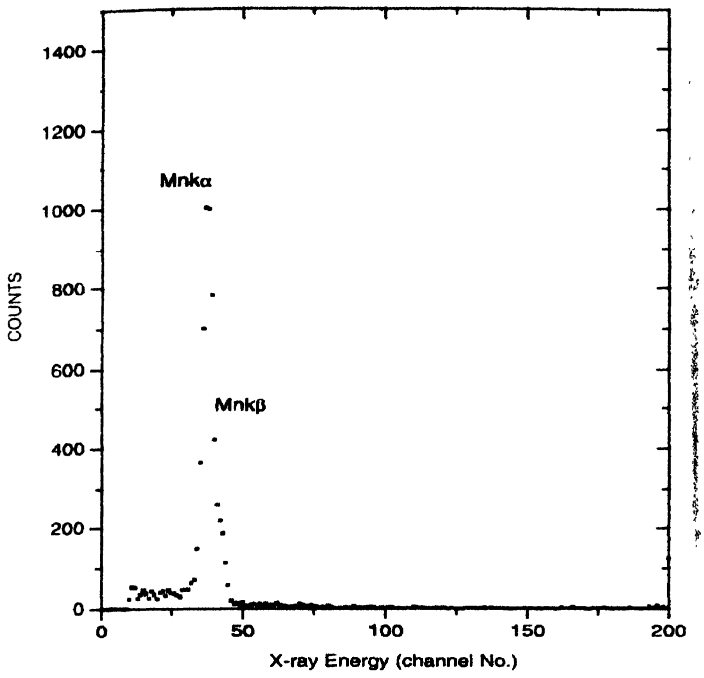

Figure 3c. PIXE X-ray spectrum of Mn standard solution.

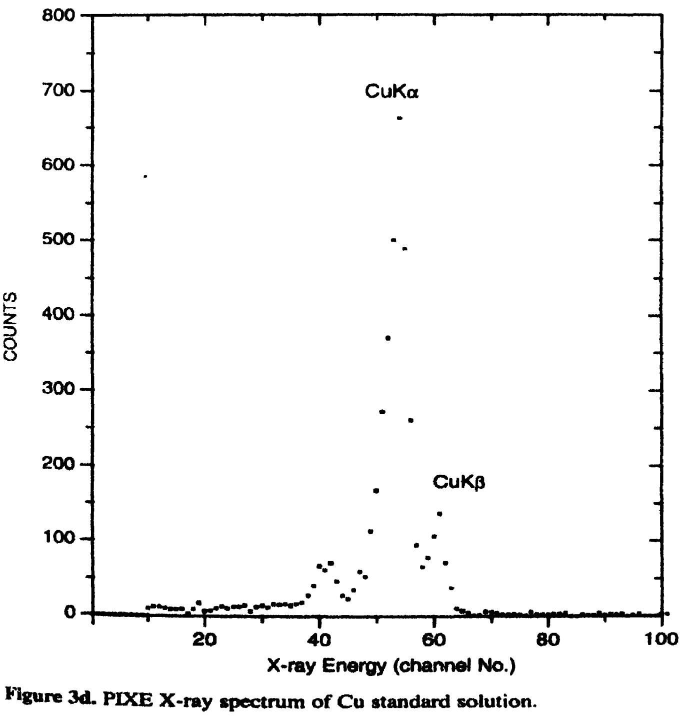

Figure 3d. PIXE X-ray spectrum of Cu standard solution.

in Be window of the detector and for the ionisation cross sections calculations. The percentage values of gold and silver were obtained by using the thick sample code (10) in which it is assumed that the major element of the

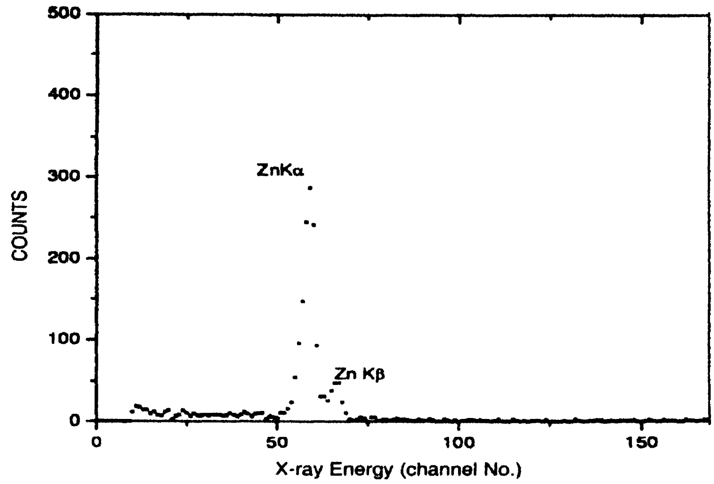

Figure 3e. PIXE X-ray spectrum of Zn standard solution.

Table 1. Percentage compositions of Au and Ag in gold standards by PIXE.

Gold standards	Au%, Ag% (PIXE)	Au%, Ag% (certified)
22 kt	94.6% ± 0.09	91.66%,
	5.4% ± 0.40	8.34%
20 kt	84.9% ± 0.06	83.33%,
	15.06% ± 0.15	16.67%
18 kt	76.73% ± 0.04	75%,
	23.26% ± 0.08	25%
4 kt	56.06% ± 0.07	58.3%
	43.93% ± 0.08	41.7%

Table 2. Detection limits of elements with 20 > Z > 30 by PIXE.

Elements	Atomic No.	Detection limit (μg/g)
Cr	24	4
Mn	25	0.8
Fe	26	2.13
Co	27	2.08
Cu	29	3.6
Zn	30	3.0

sample matrix is taken as internal standard. Assuming the matrix to be equivalent to this element, the concentrations of the other elements are computed. The formula for the calculation of weightage of the detected elements is as follows.

The relation between the X-ray intensity of a particular element to its concentration for thick sample is given by

$$I_j = I_0 G^* K_j^* r_j^* m_j / (\mathrm{cosec}\,\varphi^* \mu_{\mathrm{int}} + \mu_{\mathrm{fl},j}), \qquad (1)$$

where

$$\mu_{\mathrm{int}} = \Sigma \mu_j^* m_j / \Sigma m_j,$$

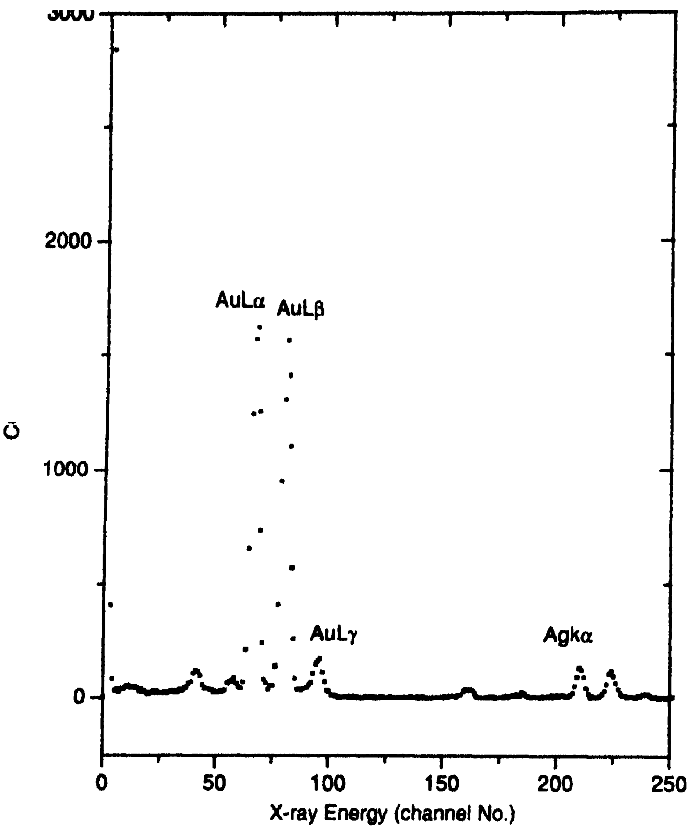

Figure 4. PIXE X-ray spectrum of 22-carat gold standard.

$$\mu_{\mathrm{flt},j} = \sum^{j} {}^{*} m_k \Big/ \sum^{j} m_k \,,$$

where

I_j = intensity of the j-th element,

Kj = relative excitation cum detection factor,

φ = average angle between the incident radiation and the surface of the sample,

μ_j = mass absorption coefficient of the j-th element at the incident radiation,

$\mu_{k,j}$ = mass absorption coefficient of the k-th element at the characteristic X-ray energy of the j-th element,

μ_{int} = weighted mass absorption coefficient at the incident radiation,

$\mu_{\mathrm{flt},j}$ = weighted mass absorption coefficient at the fluorescent energy of the j-th element.

Substituting μ_{inj}, $\mu_{\mathrm{flt},j}$, r_j, k_j, I_j in (1) I_0G is calculated. Using I_0G, r_j for other elements are calculated. The r_j's obtained are used for calculating new values of μ_{int}, $\mu_{\mathrm{flt},j}$. The iteration is continued till it converges to a certain value which does not change further. Table 1 gives the percentage values obtained by PIXE as well as XRF and the certified values. It is seen that the values are in agreement with those

4. Conclusion

The present PIXE set up can detect % values of metals in alloys and trace elements in thin targets of low-Z elements down to a level of few μg. PIXE can be applied to materials modification studies and can detect trace elements in thin targets in solution form.

Acknowledgments

We are grateful Dr. S Kailas, Nuclear Physics Division, BARC for fruitful discussions and Shri Shaji Kumar, Analytical Chemistry Division, BARC for providing the standard solutions. Acknowledgements are due to the Shri S K Gupta and the operating staff of FOTIA accelerator for smooth running of the machine.

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